STUDY OF THE CONTRACTION OF THE LATTICE PARAMETERS OF SILVER NANOPARTICLES SYNTHESIZED USING ACHYRANTHES ASPERA L STEM EXTRACT

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ABSTRACT

Achyranthes Aspera L Stem Extract has been used as a reducing and capping agent and single step green synthesis method was adopted for the process of obtaining silver nanoparticles(AGNP). The UV-visible spectrophotometer which has revealed that the surface plasmon resonance occurs at 420 nm to 450 nm due to the presence of silver nanoparticles. The nanoparticles were found to be approximately spherical in shape and their size obtained through Scanning Electron Microscope (SEM) was found to be between 30 – 80 nm, energy dispersive X-ray (EDX) analyses has shown that these nanoparticles are crystalline in nature. The characterization data was used to evaluate the surface energy of the silver nanoparticles at room temperature which was helpful in the explanation of the contraction of the lattice parameters of silver nanoparticles with decreasing particle size and they have been found to be size and shape dependent which was in agreement with the experimental results.

KEYWORDS: Lattice Parameters, Silver Nanoparticles, Green Synthesis, Characterization, Surface Energy, Mathematical Model

INTRODUCTION

The difficulties that humans are facing to organize themselves in a non-chaotic way has been a complex problem in comparison to and the ease with which nanoparticles(NPs) in general and silver nanoparticles(AGNPs) are being synthesized cinching them with the use of ethnobotanical species[1-7]. Extracts of ethnobotanical species are being utilized in the synthesis of silver nanoparticles. Intensive work has been in progress for quite a long time now and many ethnobotanical species that bear valuable medicinal properties have been successful in reducing and capping the noble metal nanoparticles and currently, the methods of production of nanoparticles is being made possible through single step techniques. These procedures have gained prominence and are being considered as a large scale substitute to the eco-hazardous production techniques due to the achievability of the size, distribution, retaining the morphological structure. Researchers identified that bio-molecules like proteins, phenols and flavonoids not only aid the ions in the Nano-size synthesis but also in shaping them[8-10]. This procedure has been identified to be Bio-convivial and viable with an ease of escalation to the mass supply needs in research and medicine. Nanoparticles ranging from a nanometer to a few hundreds of nanometers have been synthesized and characterized and were proved to possess unbound appositeness in different territories of physics, chemistry, biology, and medicine for applications in optics, catalysis, photonics, biomedicine and antimicrobial activity[11-16].

It had been observed that the study of the lattice parameter variation of the nanoparticles thus synthesized plays a vital role in the conceptual understanding where lattice parameter measurements areused in the abstraction of physical

properties [17]. The knowledge ofthe lattice parameters provides valuableinformation on the elastic, thermal and dielectric propertiesof materials, an indirect method todetermine the colloid composition,, measurement of the state of strain,or defect structure analysis*etc.,*.

This led to the current work where the size and shape dependent lattice parameters of AGNPs obtained through green synthesis using Achyranthes Aspera L stem extract had been studied.Taking the cue that the surface energy will contract the nanoparticle elastically, and relating the elastic energy with the surface energy of the nanoparticles per unit area, the effective increased surface energy has been evaluated and from the study of the variation of the total energy and the shear modulus of the bulk material, the contraction of the lattice parameters of silver nanoparticles has been evaluated and compared with the experimental results of the AGNPs obtained through green synthesis using Achyranthes Aspera L stem extract [18-22]. The current model discussed is a continuous media model and can deal with the nanoparticles up to about 100nm and the model has been found to be applicablein predicting the lattice variation of other metallic nanoparticles as well with different lattice structures as the selected parameters used in the evaluation are material's physical constants.

Synthesis and Characterization of Silver Nanoparticles

Achyranthes Aspera L

Traditional medicine from herbs has been a rural remedy for generations now constituting a major part of treatment [23]. Herbal medicine has been a triumph of popular therapeutic diversity. Plants above all other agents have been used for medicine from time immemorial because they have fitted the immediate personal need, due to their easy accessibility and economic viability [24].

Achyranthes Aspera L[25-27] is an important medicinal herb that grows as a weed throughout India. Traditional systems of medicine use all of its parts. Wide numbers of phytochemical constituents have been isolated from this plant which possesses activities like antiperiodic, diuretic, purgative, laxative, antiasthmatic, hepatoprotective, anti-allergic and many more medicinal properties. It has been used for a longtime now in the rural parts of India to cure pneumonia, the infusion of the root is used as mild astringent in bowel complaints. For the last few decades or so, extensive research work has been in progress to prove its biological activities and pharmacology of its extracts. Saponins, oleonolic acid, dihydroxy ketones, alkaloids, long chain compounds and many other chemical constituents have been isolated.

Experimental Procedure

In the single step green synthesis, the stem extract and 1 mM aqueous $AgNO_3$ solution were added in the ratio of 1:19 and heated up to 75°C for a few minutes until the capping and reduction of AGNPs was complete, which was be evident through change in the colour of the colloid. The AGNP solution had been centrifuged (10000 rpm for 15 minutes) and the supernatant was transferred into a dry beaker and stored. The sample had been dried in an incubator and the particles obtained were used for further characterization.

Characterization

UV-Vis, EDX, XRD and SEM Studies confirmed the bio-reduction of aqueous silver ions to silver nanoparticlesand the results have been published elsewhere by the same authors[28].Initially it has been confirmed by the colour change to an yellowish brown colour and later through the observation of the excitation in surface Plasmon vibrations by absorbance in

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between 420 and 450 nm using an UV-Vis spectrophotometer. The most important feature of optical absorbance spectra is the surface Plasmon band among metal nanoparticles, which is due to the electron oscillations that collectively gather around the surface of silver particles.

The presence of the elemental silver had been observed in the graph obtained from EDX analysis, which was also supported by the XRD results thus establishingthe evidence of the crystalline nature and structure of AgNPs when the sample was analyzed on X-ray powder diffractometer (XRD) using a diffractometer equipped for CuK$_\alpha$radiation (λ = 1.5418ºA) in the 2Θ range of 0-85° with a step size of 0.04° and a scanning rate of 5.0°/min. The comparison of XRD spectrum results with standards confirmed the crystalline nature of silver nanoparticles forming peaks at 2Θ values corresponding to 111, 200, 220 and 311 of Bragg reflections for silver metal. The crystallite size of AgNPs calculated by using Debye-Scherer's formula (1) had been observed to possess an average size 27 nm from XRD data and the use of Debye-Scherer equation.[28]

$$D = K\lambda/\beta\cos\theta \tag{1}$$

Where, D is the crystal size of AgNPs, $\lambda$is the wavelength of x-ray source used (1.541 Å), β is the full width at half maximum of the diffraction peak, K is the constant of Debye-Scherer equation with value from 0.9 to 1 and Θ is the Bragg angle [29]. The structural peaks present in XRD pattern and crystalline size suggested that the biosynthesized AgNPs had a crystalline nature.

Evaluation of Specific Surface Area Using XRD Data

The information about the phenomenon of absorption, heterogeneous catalysis and reactions on surfaces can be predicted through the measure of the surface area per unit mass called as the Specific Surface Area (SSA) which is a material property. It is a derived scientific value that can be used to determine the type and properties of a material.

$$SSA = \frac{SA_{part}}{V_{part} * density}$$

where V_{part} is particle volume and SA_{part} is the surface area of the particle [30].

$$S = \frac{6 * 10^3}{D_p\,\rho}$$

where S is the specific surface area, D_p is the size of the AGNPs, and ρ is the density of silver 10.5 g/cm^3 [31]. Its value was calculated from the characterization data as 46.8m^2/gm.

SEM analysis has been used to provide information about the morphology and size of the synthesized silver nanoparticles. The silver nanoparticles have been characterized using a Scanning Electron Microscope facility provided by Osmania University, Hyderabad, Telangana, India. The SEM results revealed that the average size of AGNPs synthesized were found as to be nearly spherical in shape and 30-80nm in size.

Model to Study the Contraction of the Lattice Parameters

It has been reported that the lattice parameters of nanoparticles depend on their size and shapeand the same has been verified experimentally[32-36]. In these studies the NPs have been regarded as ideal spheres and the same has also been reported for the non-spherical NPs [37-39]. The lattice parameter variation of NPs is a surface effect phenomenon

which requires the particle shape to be taken into consideration.[18-22].

In developing the mathematical model, initially a particle from the bulk has been isolated assuming no loss of the structural properties. The surface tension of such an isolated particlewould contract its size elastically, and finally aids itsformation in an equilibrium state tending to attain a shape that possesses a minimum energy. A shape factor α [19] has been introduced to include non-spherical NPs. By minimizing the sum of the increased surface energy and the elastic energy, a formula has been obtained in the generation of the lattice parameters. Theselattice parameters have been compared with the corresponding experimental values.

The shape factor has been defined as follows[20]

$$\alpha = \frac{A'}{A} \quad (or \ A' = \alpha A) \tag{1}$$

Where $A = A'.4\pi R^2. \rightarrow$ surface area of a spherical NP and A' is the surface area of the nanoparticle of any shape and same volume. For spherical nanoparticle, we have α=1, and for non-spherical nanoparticle, α>1.

The increased surface energy after the NPs have been isolated from the crystal , where Δγ is given by

$$\Delta\gamma = \alpha. \ 4\pi R^2 \gamma \tag{2}$$

where, R is the radius of the particles,

γ is the surface energy per unit area at the temperature T

And (0 ≤T <T$_m$, T$_m$)is the melting temperature of metals).

γhas been obtained from the equation given below

$$\gamma = \gamma_0 + T\frac{d\gamma}{dT} \tag{3}$$

where, γ$_0$ is the surface energy per unit area at 0 K,

The surface energy would contract the nanoparticle elastically and this the surface energy per unit area is independent of the particle size [40]and dγ/dT is the coefficient of surface free energy to temperature. For most solids, dγ/dT < 0 [44]

Assuming a small displacement ϵR resulting from this elastic contraction, where $\epsilon \ll 1$.

For spherical particles, the elastic energy E'_ecan be written applying the theory of elasticity as

$$E'_e = 8\pi\sigma R^3 \epsilon^2 \tag{4}$$

where σ is the shear modulus. As the metallic nanoparticles have been assumed to be formed out of infinitesimal elastic contractions after separation from the bulk the use of shear modulus values corresponding bulk metals is justified.

Considering the expression $A = 4\pi R^2$,

$$E'_e = \pi^{-\frac{1}{2}}\sigma A^{\frac{3}{2}}\epsilon^2 \tag{5}$$

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Where, the parameter ϵ is a variable, which can be regarded the same for nanoparticles in any shapes. And equation (5) has been modified by incorporating the shape aspect from equation(1) into (5)for NPs of non-spherical shape,

$$E_\epsilon = \alpha^{\frac{3}{2}}\pi^{-\frac{1}{2}}A^{\frac{3}{2}}\sigma\epsilon^2 \tag{6}$$

Equation (6) can be rewritten as[39]

$$E_\epsilon = \alpha^{\frac{3}{2}}.8\pi.\ R^3.\sigma.\epsilon^2 \tag{7}$$

The contraction would decrease the increased surface energy, andthe effective increased surface energy would then be

$$\Delta\gamma = \alpha.\,4\pi\,[R(1-\epsilon)]^2\gamma \tag{8}$$

The total energy variation = Increased surface energy + Increased elastic energy, substituting and simplifying the Eqn.(4) through Eqn.(8) yields a quadratic equation of the form

$$E_\epsilon = L\epsilon^2 + M\epsilon + \text{N} \tag{9}$$

Where the values of L, M,N are respectively - $L = 4\pi\gamma R^2\alpha + 8\pi\sigma R^3\epsilon^2$, $M = -8\pi\gamma R^2\alpha$, $N = 4\pi\gamma R^2\alpha$

Minimizing the energy at equilibrium results in$\frac{dE_\epsilon}{d\epsilon} = 0$and

$$\epsilon = \frac{1}{1+\left(\frac{2\sigma}{\gamma}\right).R.\alpha^{\frac{1}{2}}} \tag{10}$$

For an ideal crystal lattice, the lattice parameter contraction is proportional to the diameter of nanoparticle

$$\frac{\Delta a}{a} = \frac{(a_p - a)}{a} = \frac{(1-\epsilon)R - R}{R} \tag{11}$$

where a_p and a are the lattice parameters of the nanoparticle and the corresponding bulk material. Inserting Eq. (10) into Eq. (11), the study of the variation of the lattice parameters would be possible

$$\frac{\Delta a}{a} = \frac{1}{1+K.D} \tag{12}$$

Equation (12) is the basic relation for the size and shape dependent lattice parameters of metallic nanoparticles,

where D (= 2R) is the diameter of the nanoparticle,

and $K = \frac{\alpha^{\frac{1}{2}}\sigma}{\gamma}$.

Generally, both of the shear module and the surface energy are positive; therefore, the lattice parameter of the metallic nanoparticles will decrease with decreasein particle size.

Table 1: The Input Physical Parameters for the Silver Nanoparticles

σ Shear module (298 K) [42]	$3.03 \times 10^{10} (N/m^2)$
γ_0 Surface energy (0 K) [43]	$1.250 (J/m^2)$
$d\gamma/dT$ coefficient of surface energy to Temp. **[43]**	$- 15 \times 10^{-5} (J/(K \, m^2)$
γ Surface energy (298 K) (From Eqn.(3))	$1.205 (J/m^2)$

RESULTS AND DISCUSSIONS

Since the lattice parameters vary with the size and the shape of nanoparticles, the particle size effect on lattice parameter has been evaluated for the spherical and nearly spherical AGNPs obtained experimentally from the Achyranthes Aspera stem extract.

To calculate the lattice parameters of metallic nanoparticles by Eq. (12), it is needed to determine the values of shape factor. And standard values of the shape factor [41] have been adopted - (Spherical – 1, Regular tetrahedral - 1.49, Regular hexahedral-1.24, Regular octahedral-1.18, Disk-like- >1.15, Regular quadrangular- >1.24).The shape dependent lattice parameters of nanoparticles in specific size have been simulated using MATLAB program and the results have been presented below.
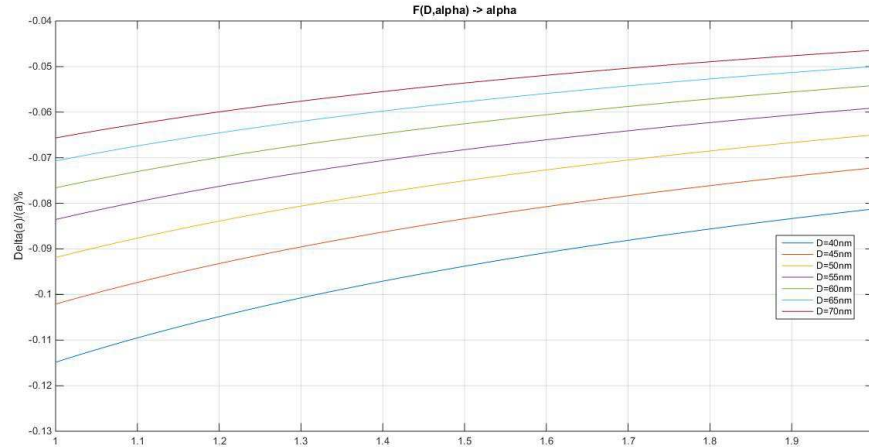
Figure 1: Variation of the Relative Parameter of AGNPs as a Function of Shape Factor

(Results calculated from Eq. (12).)

For AGNPs with 40 nm, the lattice parameter variation equals -0.83% for spherical shape, but -0.70% for regular tetrahedral shape; for AGNPs with 50 nm, the lattice parameter variation equals -0.48% for spherical shape, but -0.34% for regular tetrahedral shape; similarly, for AGNPs with 80 nm, the lattice parameter variation equals -0.18% for spherical shapes, but -0.14% for regular tetrahedral shape.

Limitations

This model is not applicable for systems of Nanowires and Nano-films as the shape factor is unbound. (for

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nanowires and nano-films, the shape factor approaches to infinity).

The applicability of this model in the case of Doped Nano-crystals where the results have been contrary is out of scope of the current study and has been reported elsewhere [44-47].

The results have led to the following discussion:

- The variation of the lattice parameter has been found to decrease with the increase in the shape factor, which in turn depends on the particle dimensions.

- It has been identified that the dependence of lattice parameter on the shape has been stronger in the case of smaller particle sizes and it plays a greater role in the variation.

- The particle dimensions have been found to contribute about 10-15% to the total lattice variation, which would result in the same relative error if this aspect is ignored during the calculation of lattice parameters of metallic nanoparticles.
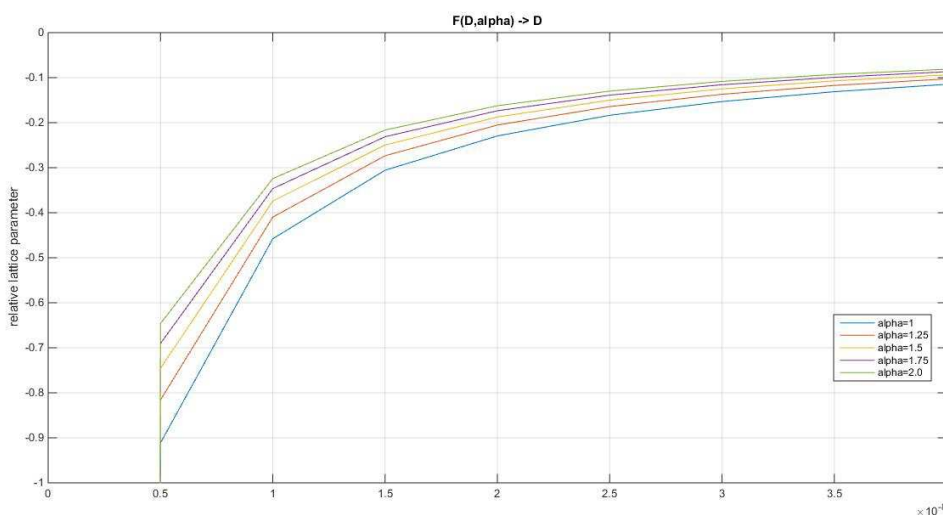
Figure 2: Variation of the Relative lattice Parameter as the Function of the Diameter of AGNPs

- As the model has been successful in bringing out a definitive relation to the experimental results to that of the theory with which many new characteristics of the nanostructures can be understood, it stands out as a 'continuous media model' and can be used to predict the lattice parameter variation of metallic nanoparticles.

- In the non-spherical case of nanoparticles where in spite of a different shape theycould still possess the same value of the shape factor the description is approximate as it is defined by the surface area.

CONCLUSIONS

A Mathematical model that was successful in accounting for the size and shape dependent lattice parameters of metallic nanoparticles, where the particle shape difference has been considered by introducing a shape factor.

It is predicted that the lattice parameters of AGNPs in several nanometers decrease with decreasein the particle size, which is well consistent with the experimental results.

Furthermore, it has been observed that the particle shape would lead to about 10-15% of the total lattice variation depending on the particle shape.

The model used is a continuous media model and can deal with the nanoparticles of size ranging between 1nm to about 90nm, above which the accuracy deters .

The values of the physical constants like, shear module, surface energy per unit area, standard shape factors of the crystal lattices of the corresponding bulk material are easily available and hence this model can be adopted to study and extrapolate the variation of lattice parameters in the preliminary analysis of any research work on green synthesis of crystalline metal nanoparticles.

Finally, this model may be widely used to predict the lattice parameter variation of small metallic particles, and it is being explored further to study many other associated complex characteristics in nanophysics such as variation of dielectric and elastic properties and their influence in the controlled synthesis of nanoparticles.

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